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PROPERTIES OF NONAQUEOUS ELECTROLYTES

SIXTH SUMMARY REPORT

(20 September 1967 to 19 March 1968)

By

Rudolf Keller, James N. Foster, Douglas C. Hanson, John F. Hon,
Otto F. Kalman, James S. Muirhead, and Jack M. Sullivan

Prepared For

National Aeronautics and Space Administration

Contract NAS3-8521

Rocketdyne
A Division of North American Rockwell Corporation
Canoga Park, California

NOTICE

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FOREWORD

This report was prepared under G.O. 08852 in compliance
with Article VI and Paragraph B of Contract NAS3-8521
for the National Aeronautics and Space Administration,
Lewis Research Center, Cleveland, Ohio. The work was
conducted in the Chemical and Material Sciences Depart-
ment of Rocketdyne's Research Division, during the
period 20 September 1967 through 19 March 1968.

ABSTRACT

Studies of aprotic electrolytes based on three solvents:
propylene carbonate, dimethyl formamide, and acetonitrile,
were continued. Characterized components were used to
prepare the electrolyte solutions.

Structural studies of electrolytes containing lithium
chloride, aluminum chloride, and cupric chloride were
performed utilizing nuclear magnetic resonance.

The physical properties studied include solubilities,
vapor pressures, diffusion coefficients, and dielectric
constants.

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ILLUSTRATIONS

TABLES

SUMMARY

Physical properties and structural studies were conducted in the solvents propylene carbonate (PC), dimethyl formamide (DMF), and acetonitrile (AN); the water content of the solvents was normally in the range of 40 ±20 ppm.

The distilled solvent batches were analyzed by vapor-phase chromatography on a routine basis. Some solute materials such as CuF_2, BF_3, and PF_5 were analyzed. Sensitivity tests of concentrated lithium perchlorate solutions were negative.

High-resolution proton NMR and broadline Al^{27} NMR data have been analyzed for both 1 M $AlCl_3$/AN and 1 M $AlCl_3$/PC specimens containing various concentrations of LiCl. This analysis indicates that the major species formed when $AlCl_3$ is dissolved in both AN and PC are, respectively, $AlCl_4^-$ and a sixfold coordination complex, $Al(AN)_6^{+++}$ or $Al(PC)_6^{+++}$. Broadline Cl^{35} data indicate also the presence of small concentrations of Cl^- in all of the AN specimens.

High-resolution proton NMR spectra were taken over a 6-month period of 1 M $CuCl_2$/DMF specimens containing various concentrations of LiCl. Long-term changes in these spectra have been noted and have been followed. Thus far, the data have not been explained conclusively; however, they have shown the need for a revision in the interpretation of the early data.

Solubilities of CuF_2 in propylene carbonate and dimethyl formamide were found to be low in the pure solvents, but enhanced in the presence of lithium salts, evidently because of the formation of insoluble lithium fluoride.

Vapor pressures for various solutions were measured at 25 and 60 C by the saturation method. Diffusion coefficients of some dimethyl formamide solutions were determined at 25 C by a porous disk method. The determination of diffusion coefficients of copper species by chronopotentiometry was studied. Preparatory work for the measurement of heats of solution and dielectric constants was essentially completed.

DESCRIPTION OF PROGRESS

PREPARATION OF ELECTROLYTES

Purification of Solvents

Solvents were purified by distillation and analyzed as described previously (Ref. 1 through 5). The normally applied analytical procedure and distillation conditions were summarized in Ref. 3. The purification procedure for DMF was somewhat modified. Most batches were distilled with a 1-foot Vigreux column at atmospheric pressure. CaH_2 was used as the drying agent, but its use has been discontinued because of the possibility of a side reaction producing water. The procedure used most recently involves a predrying with molecular sieves and a distillation of the centrifuged predried DMF.

The solvent batches used during the report period are listed in Table 1. Some batches containing more water than 40 ±20 ppm were used in some exceptional cases, e.g., in diffusion coefficient measurements where the water content of the bulk solvent does not appear to be critical.

Analysis of Cupric Fluoride

A cupric fluoride sample, CuF_2 #2 (Ozark-Mahoning Company, special quality), had been analyzed previously (Ref. 4). The analysis of a more recently obtained product, CuF_2 #3 (Ledoux & Company, special quality), is presented in Table 2. Low impurity levels for metallic impurities were recorded. A large oxygen content of 1.5 percent by weight was revealed by spark source mass spectrometry, together with significant amounts of carbon and some sulfur. In the second analysis, which was provided by the supplier of the chemical and was performed by the Associated Electrical Industries, Ltd., England, no figures for O, N, and C are given; nitrogen and oxygen could not be determined because of instrument background, and in the case of carbon, a determination was impossible because the sample was mixed with graphite.

TABLE 1

CHARACTERIZATION OF DISTILLED SOLVENT BATCHES

Solvent Code	H_2O Content, ppm	Organics, ppm
PC #2-11	32	None
PC #2-12	33	None
DMF #5-1	52	26
DMF #5-2	65	9
DMF #5-3	38	None
DMF #5-4	56	None
DMF #5-5	57	None
DMF #5-6	52	None
DMF #6-1	59	None
DMF #6-2	30	None
DMF #6-3	74	None
DMF #6-4	109	None
DMF #6-5	75	None
DMF #6-6	150	None
DMF #6-7	155	None
DMF #6-8	110	None
DMF #6-9	77	None
DMF #6-10	48	None
AN #4-2	60	None

The high oxygen content appears to be due to the presence of a copper oxide rather than an excessive water content because only small amounts of hydrogen were found. CuF_2 #3 is being used for measurements despite the relatively high impurity levels; a more satisfactory product does not seem to be available at the present time.

Analysis of Tetramethylammonium Fluoride

Two tetramethylammonium fluoride products were analyzed (TMA·F #2, Aldrich Chemical Company, Inc., and TMA·F #3, Southwestern Analytical Chemicals, Inc.). The results of an emission spectrographic analysis performed by Pacific Spectrochemical Laboratory, Inc. are presented in Table 3. Impurity contents not listed are below the detection limits listed in Ref.1 . No results could be obtained by spark source mass spectrometry because the sample lost its consistency upon exposure to vacuum and could not be sparked; TMA·F undergoes thermal decomposition to $(CH_3)_3N$, CH_4, and CH_3F in a vacuum of 0.5 to 1 mm Hg according to Ref. 6, and a decomposition could be expected at room temperature at a vacuum of 10^{-7} to 10^{-8} mm Hg as in the mass spectrometer.

In a visual examination of the two products, TMA·F #2 was found to have the appearance of a dry powder, whereas TMA·F #3 was more cakey and a relatively high water content of this later product may be suspected. However, TMA·F #2 was rejected because of the very high sodium content. Southwestern Analytical Chemicals, Inc. is manufacturing an improved product (Ref. 7) and a sample has been ordered.

Analysis of Boron Trifluoride

A cylinder of boron trifluoride (BF_3 #1, Matheson, C. P. Grade) was analyzed using a CEC 21-103C mass spectrometer. Trifluoroboroxime was the only impurity found. Its concentration was determined to be greater than 1 mole percent. Although the mass spectrometer was extensively

TABLE 2

IMPURITY CONCENTRATIONS IN CuF_2 #3 DETERMINED BY

SPARK SOURCE MASS SPECTROMETRY AND EMISSION SPECTROSCOPY

Element	Spark Source Mass Spectrometry (by Bell & Howell)			Spark Source Mass Spectrometry (by Associated Electronic Industries)			Emission Spectroscopy (by Pacific Spectro-chemical Laboratory)	Emission Spectroscopy (by Ledoux & Company)
	Detection Limit, ppm atomic	Impurity Concentration, ppm — Atomic	Impurity Concentration, ppm — Per Weight	Detection Limit*	Impurity Concentration, ppm — Atomic	Impurity Concentration, ppm — Per Weight	Impurity Concentration, ppm per Weight	Impurity Concentration, ppm per Weight(?)
H	3.0	280	8.3					
Li	0.3	0.3	0.06					
Be	1.0	15	4					
B	1.0	14	4		1	0.3		
C	1.0	1500	500					
N	1.0	26	11					
O	1.0	32,000	15,000					
Na	0.3	71	48		2	1	2.7	8
Mg					5	4		3
Al	0.5	46	37		5	4	<5	3
Si					20	17		
P					1.5	1.4		
S	5.0	240	230		10	10		
Cl	2.0	93	97					
K	0.3	36	42		3	3	6.9	5
Ca	0.7	8.5	10		4	5		
Ti	7.0	<7	<10		0.2	0.3		
Cr	1.0	6.1	9.4		0.3	0.5		
Mn					4	7	<2	5
Fe								5
Co	2.0	4.0	7.0		2	3		
Ni	2.0	4.9	8.5					
Zn	5.0	<5	<10					
Ga	0.7	5.5	11					
As	0.7	31	69					
Rb	0.7	2.0	5.0					
Te	10.0	<10	<38					
Pb					5	30		

*Not available

TABLE 3

IMPURITY CONCENTRATIONS (ppm by weight) IN TMA·F
DETERMINED BY EMISSION SPECTROGRAPHY

Impurity \ Solute	TMA·F #2	TMA·F #2*	TMA·F #3
B	1200	12,000	
Mg	260	210	5.0
Al	1800	33	
Si	4100	3500	53
Na	250,000	180,000	
Ca	400	590	25
Ti	1200		
Cr	190		
Mn	47	9	
Fe	150	29	
Ni			
Cu	10	6.5	5.2
Ag	150		
Sn	250		

*Second batch of same order, deliver 2-1/2 months
after first batch.

Impurity contents of elements not listed are below detection limits
listed in Ref. 1.

passivated, the $(BOF)_3$ found may not be present in the sample, but may instead be generated inside the mass spectrometer. Fisher, Lehmann, and Shapiro (Ref. 8) report that $(BOF)_3$ is not stable at temperatures below 250 C, and that a sample of $(BOF)_3$ is essentially decomposed (>99 percent) after 1 hour at 25 C. Thus, the $(BOF)_3$ found in this analysis must have been generated in the mass spectrometer and is not actually present in the BF_3 sample. Permanent gases, such as SiF_4, CF_4, N_2, O_2, and SF_6, were not detected and, consequently, were present at concentrations less than 0.1 mole percent, which is the detection limit for these species (fluorine cannot be detected mass spectrometrically in the presence of large amounts of boron trifluoride).

The BF_3 was also analyzed by gas chromatography using a Halocarbon Oil 13-21 on a Chromasorb W column but the results were inconclusive; peaks were found but they were not reproducible, indicating that the sample was incompatible with the packing or the column had not been completely passivated.

The infrared spectrum of BF_3 in a 5-centimeter cell with silver chloride windows containing 80 millimeters of BF_3 was recorded from 670 to 3500 cm^{-1} on a Perkin Elmer Infracord, Model 137. No peaks were found other than those expected for BF_3 (Ref. 9). Hydrogen fluoride was determined by measuring the absorbance at 3878 cm^{-1} on a Cary 14 recording spectrometer (a calibration curve was prepared by measuring the absorbance of hydrogen fluoride at various pressures).

In a first determination, using a 10-centimeter cell with calcium fluoride windows at a sample pressure of 653 millimeters, a peak was found for hydrogen fluoride, but the peak height was approximately the same as the baseline noise and was at the limit of detection. The determination was repeated using a 10-centimeter cell with sapphire windows and a sample pressure of 1495 millimeters. The peak height again was on the order of the baseline noise. The actual HF content was therefore at the limit of detection, 200 ppm, or less.

Analysis of Phosphorous Pentafluoride

The only impurity found in phosphorous pentafluoride (PF_5 #1, Research Organic Chemical Company, C.P. Grade) by mass spectrometry was POF_3, its concentration being greater than 1 percent. As in the case of BF_3, the oxygen-containing impurity found may not be present in the PF_5 sample but may be generated inside the mass spectrometer. Permanent gases, such as SiF_4, CF_4, N_2, O_2, and SF_6 were not detected, and hence were not present at concentrations greater than 0.1 mole percent. PF_3 cannot be determined mass spectrometrically in the presence of a large excess of PF_5 because the major species generated by ionization of PF_3, i.e., PF_2^+, is also generated by the ionization of PF_5. In addition, fluorine cannot be detected because fluoride ions are also produced by the ionization of PF_5.

The analyses of PF_5 by gas chromatography using a column of Halocarbon Oil 13-21 on Kel F was inconclusive. Peaks were obtained but they were not reproducible, indicating incompatibility with column packing or that the two columns had not been completely passivated.

The phosphorous pentafluoride was analyzed by infrared spectrometry to supplement the mass spectrometric results. The spectrum of phosphorous pentafluoride at 720 millimeters is shown in Fig. 1; it was recorded on a Perkin Elmer Infracord, Model 137, using a 10-centimeter cell with silver chloride windows. The absorption peak at 1420 cm^{-1} is due to POF_3 and the peaks at 860 and 890 cm^{-1} are due to PF_3. The remaining peaks are those expected for phosphorous pentafluoride (Ref. 10 and 11). A mixture of 20-millimeter phosphorous trifluoride, (Ozark-Mahoning Company, no purity given) and 240-millimeter helium was prepared for calibration purposes. The absorbance at 860 and 890 cm^{-1} was measured as a function of the partial pressure of PF_3, as shown in Fig. 2a. The PF_3 absorbance in the PF_5 #1 sample corresponds to 2.35 and 1.90 millimeters at 860 and 890 cm^{-1}, respectively, or a concentration of approximately 0.2 percent (by weight).
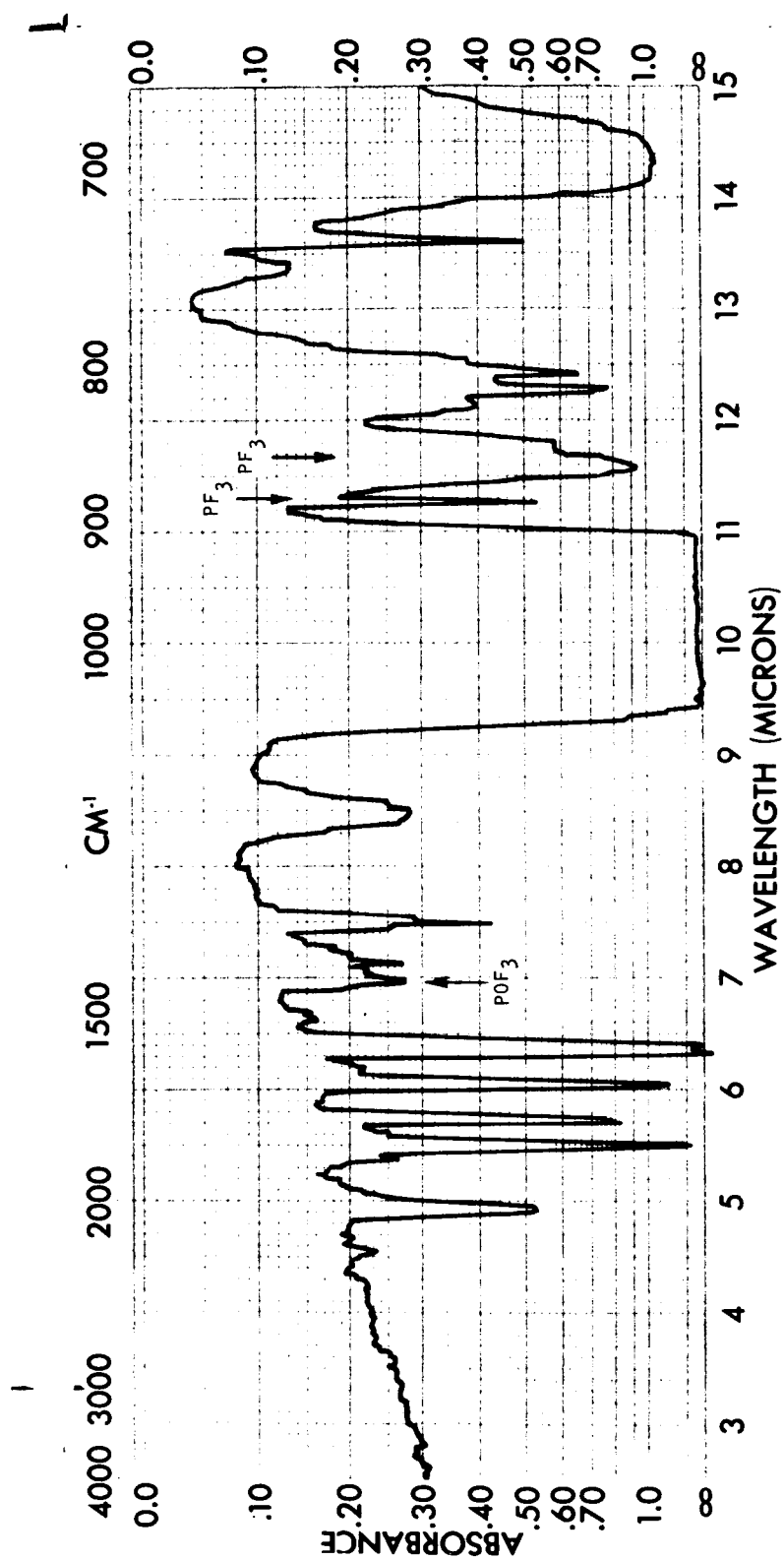
Figure 1. Infrared Spectrum of Phosphorous Pentafluoride (PF_5 #1).
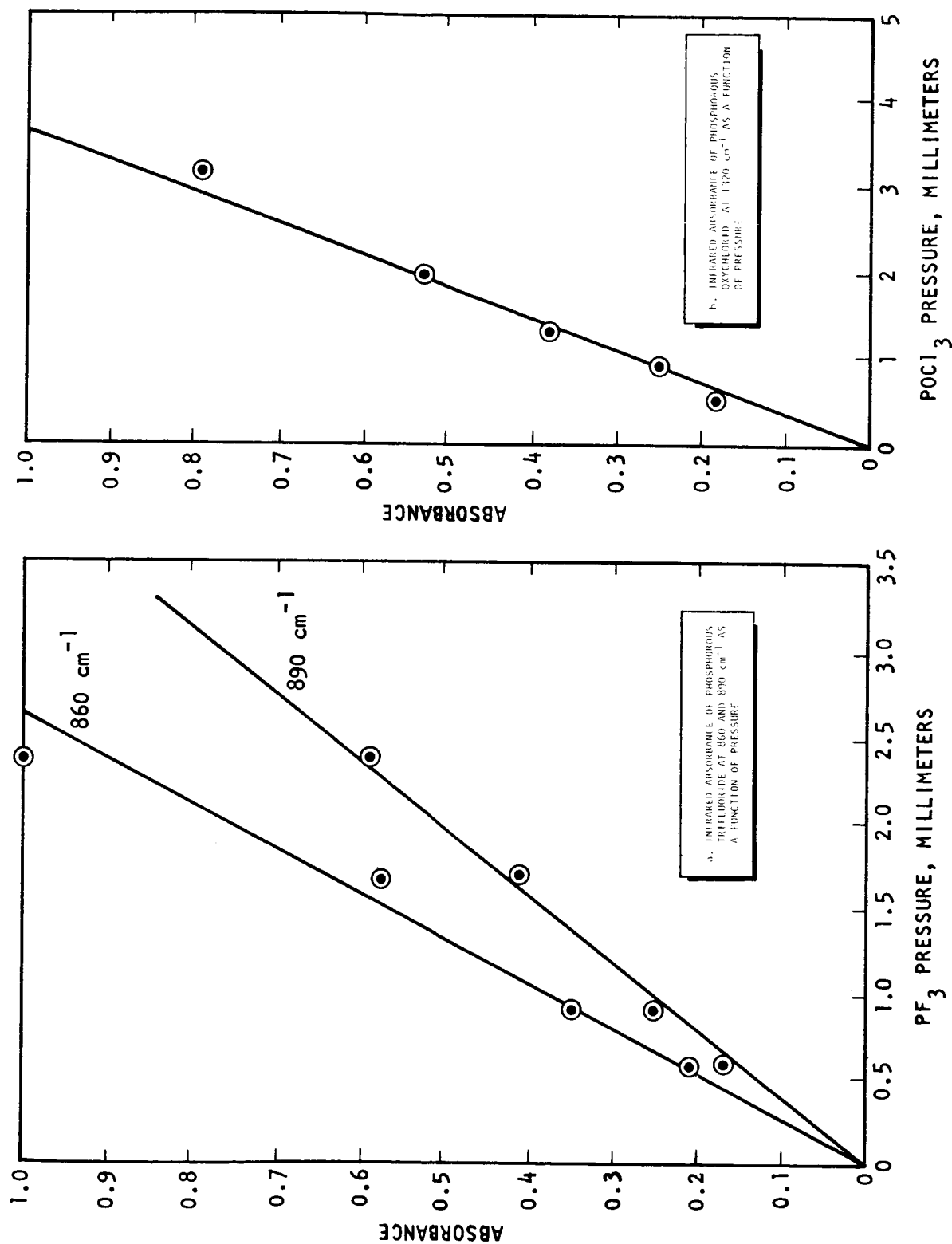
Figure 2. Infrared Absorbance of Phosphorous Trifluoride and Phosphorous Oxychloride as a Function of Pressure

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The spectrum of POF_3 has been reported (Ref. 10 and 12) but no extinction coefficients are available. The preparation of POF_3 with a known purity is very difficult and an indirect method has been employed to determine the extinction coefficient. The intense absorption peak for POF_3 at 1420 cm^{-1} is due to the stretching of the P-O bond. Assuming that $POCl_3$ and POF_3 have similar characteristics, the extinction coefficient of the P-O bond should be the same for these two species even though the absorption frequency is different due to the differences in the masses of the two species. A mixture of 13 millimeters of phosphorous oxychloride, $POCl_3$ (Baker analyzed reagent grade), and 700 millimeters of helium was prepared and the absorbance was measured at 1320 cm^{-1} at different pressures. The absorbance of phosphorous oxychloride as a function of its partial pressure is shown in Fig. 2b. The minimum absorbance peak for POF_3 in PF_5 #1 had an absorbance of 0.08 (Fig. 1), which corresponds to 0.3 millimeter of $POCl_3$ or approximately 300 ppm (by weight) of POF_3 in PF_5. This absorbance was repeatedly obtained but some spectra showing larger amounts of POF_3 were also found. The larger concentrations in these cases are probably due to the phosphorous pentafluoride reacting with traces of water. The actual POF_3 concentration may actually be less than 300 ppm. The much greater value found for POF_3 by mass spectrometry is due to incomplete passivation of the inlet system.

The HF concentration in the PF_5, determined by measuring the absorbance of a 500-millimeter sample in a 10-centimeter cell with calcium fluoride windows at 3878 cm^{-1}, was 800 ppm.

Preparation of $AlCl_3$/PC Solutions

Solutions resulting from dissolution of $AlCl_3$ in PC have a tendency to discolor when being prepared, and also on standing. The dissolution reaction of $AlCl_3$ in PC is exothermic, and a strong discoloration is obtained if the solution is allowed to heat up overall or locally when the solution is prepared. It was found that only slightly tinted solution could be made by adding the solute very slowly, grain by grain, under

vigorous stirring, and this method had been used to prepare $AlCl_3$/PC solutions. Very light solutions were obtained by preparing a slurry of aluminum chloride, liquid nitrogen, and propylene carbonate and letting this slurry warm very slowly. This procedure was modified because the amount of condensed water introduced with the liquid nitrogen was unknown. In a modification of this procedure, the aluminum chloride was cooled in a volumetric flask to liquid nitrogen temperature, the solvent was added, and the mixture was allowed to thaw with repeated partial refreezing. Solutions which were only slightly discolored were obtained in this manner.

Sensitivity Testing of Perchlorate Solutions

Lithium perchlorate solutions such as are being prepared on this program are thermodynamically unstable. A potential explosive force can be calculated from thermodynamic data according to Ref. 13 (nRT-method). It has been calculated, e.g., that 2 M $LiClO_4$/DMF has 52.6 percent of the explosive force of TNT.

Sensitivity tests were performed with 2 M $LiClO_4$ #2/PC #2-11, 3.5 M $LiClO_4$ #2/DMF #6-3, and saturated $LiClO_4$ #2/AN #4-2 (less than 2 molar). These solutions were subjected to 250 in.-lb of impact on a modified Jet Propulsion Laboratory impact tester and to 72 inches by 2 pounds on an Olin Matheson drop weight tester. All responses were negative.

Although these tests seem to indicate that the above solutions can be handled safely, such sensitivity tests are never completely conclusive. The same solutions could give positive results on different types of tests; they could constitute, e.g., an explosive hazard upon heating, sparking, etc.

NUCLEAR MAGNETIC RESONANCE STRUCTURAL STUDIES

$AlCl_3$ and LiCl in Acetonitrile

It has been hypothesized previously (Ref. 5) that the major species reaction that occurs in 1 M $AlCl_3$/AN is:

$$4\ AlCl_3 + n\ AN \rightarrow Al(AN)_n^{+++} + 3\ AlCl_4^- \tag{1}$$
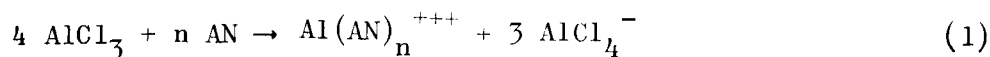
based on NMR results. Studies of the Cl^{35} resonance in several LiCl #2 + $AlCl_3$ #3/AN #4-1 solutions have revealed the presence of Cl^- ions in all of the solutions, even the 1 M $AlCl_3$/AN solution. The concentration of Cl^- ions has not been determined as yet. Several known solutions were tried for calibration purposes, primarily aqueous HCl of different concentrations; however, none was found to be suitable. The Cl^- ion concentration is apparently small compared to the concentration of Al-containing species, but not negligible, thus indicating that the addition of $AlCl_3$ to AN does not result in the completely quantitative reaction shown above.

The Cl^{35} data indicate that Eq. 1 should be expressed in an amended form:

$$4\ AlCl_3 + nAN \rightarrow \left(1 + \frac{\epsilon}{4}\right) Al(AN)_n^{+++} + \left(3 - \frac{\epsilon}{4}\right) AlCl_4^- + \epsilon\ Cl^- \tag{2}$$
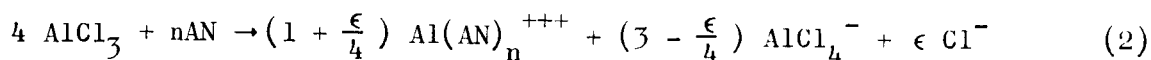
where ϵ represents the number of moles of Cl^- formed (per 4 moles of aluminum chloride). The ratio of coordinated Al to all Al is then given by $\left(1 + \frac{\epsilon}{4}\right)/4$ rather than 1/4. However, because ϵ is apparently small this ratio will still be close to 1/4.

The broadening of the AN proton lines shown and discussed in Ref. 4 has been investigated further. Spectra were taken at several temperatures from room temperature to approximately -25 C. Two of these spectra taken at 30 and -23 C are shown in Fig. 3 . As expected, both proton lines narrow at the lower temperature which is consistent with the broadening of the lines being due to exchange effects.

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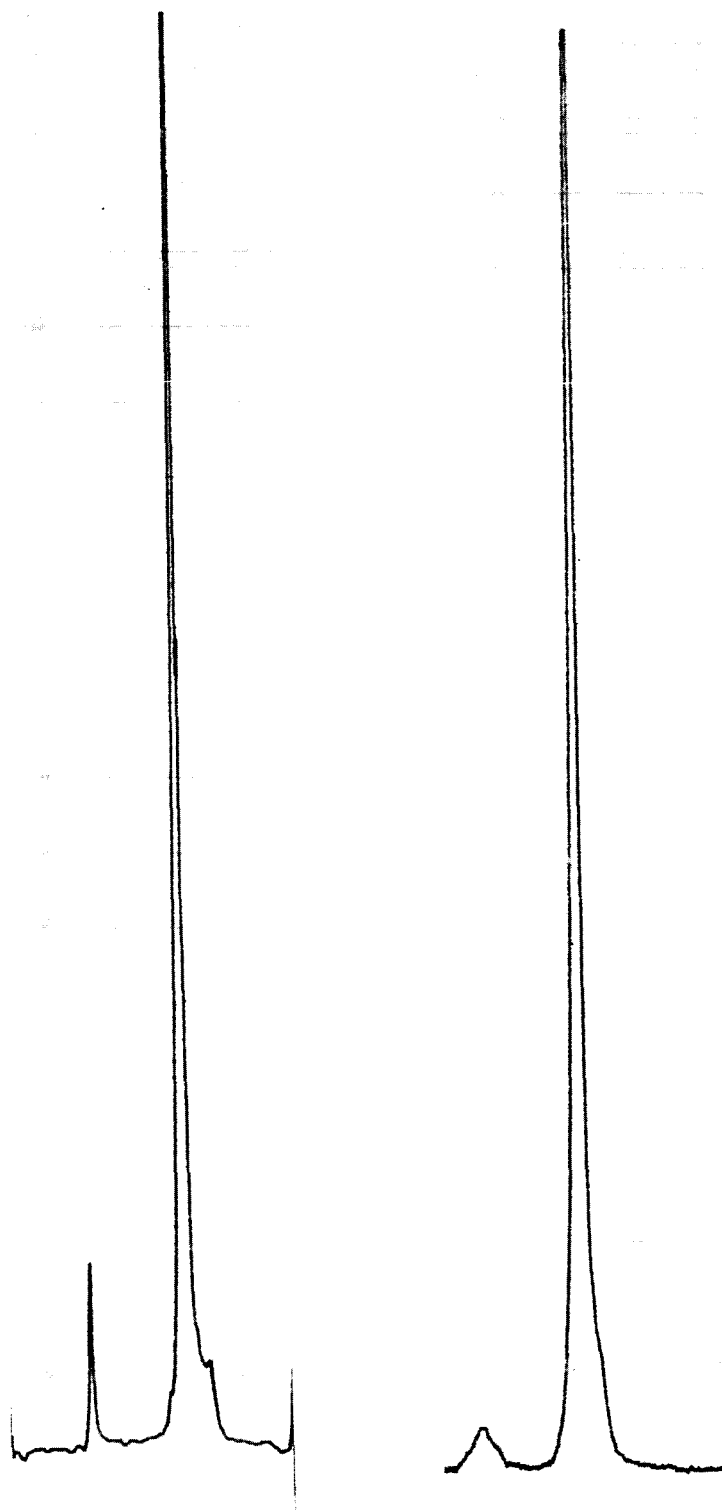
a. Low Temperature (-23 C)   b. Room Temperature (30 C)

Figure 3 .   High Resolution Proton (H^1) Resonance in 1 M $AlCl_3/AN$

To obtain n in Eq. 2, the proton lines in the AN spectra due to coordinated and bulk AN were integrated. This gives the ratio of coordinated to coordinated plus bulk AN. Utilizing the results of Ref. 5 , which substantiates Eq. 1, the ratio is multipled by four and the molar ratio of AN to $AlCl_3$. This gives a coordination number of 6.2. The limits of error in the area ratio are estimated to approximately ± 5 percent. The error in the factor of four cannot be determined but is expected to be small because ϵ (Eq. 2) is small. Thus, the coordination number for Al^{+++} in AN is determined to be 6. This is in good agreement with results for Al^{+++} in other solvents (Ref. 14 through 16).

$AlCl_3$ and LiCl in Propylene Carbonate

__Broadline Al^{27} NMR Spectra__. The Al^{27} spectra have been recorded for several 1 M $AlCl_3$ #3/PC #2-12 solutions containing different concentrations of LiCl #2 using the broadline spectrometer. These spectra are shown in Fig. 4. Chemical shift measurements using a 1 M $AlCl_3$ aqueous solution in the inner tube of a coaxial tube specimen show that the less intense line is that due to the coordinating Al containing species. These data have been analyzed in the same manner as that reported previously (Ref. 5) for the LiCl + $AlCl_3$/AN solution. A plot of the relative concentration of the coordinating Al species as a function of LiCl concentration is shown in Fig. 5 . The results are very similar to those obtained for the LiCl + $AlCl_3$/AN series. Thus, the following major species reaction occurs in the $AlCl_3$/PC solution as well:

$$4\ AlCl_3 + n\ PC \rightarrow Al(PC)_n^{+++} + 3\ AlCl_4^{-} \tag{3}$$
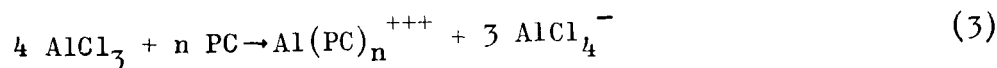
The Al^{27} spectra were obtained also for 0.1 M $AlCl_3$ #3/PC #2-12. This is shown in Fig. 6 . Because of the much lower signal (one-tenth of that for 1 M $AlCl_3$/PC) the spectra were recorded much slower with higher gain and longer time constant. The spectra show the same characteristics as those obtained from the 1 M $AlCl_3$/PC, namely, two aluminum-containing species with relative intensity of 3 to 1. From this result it appears that the aluminum-containing species present in 0.1 M $AlCl_3$/PC do not differ from those in 1 M $AlCl_3$/PC.

a. No LiCl   b. 0.25 M LiCl   c. 0.50 M LiCl   d. 0.70 M LiCl
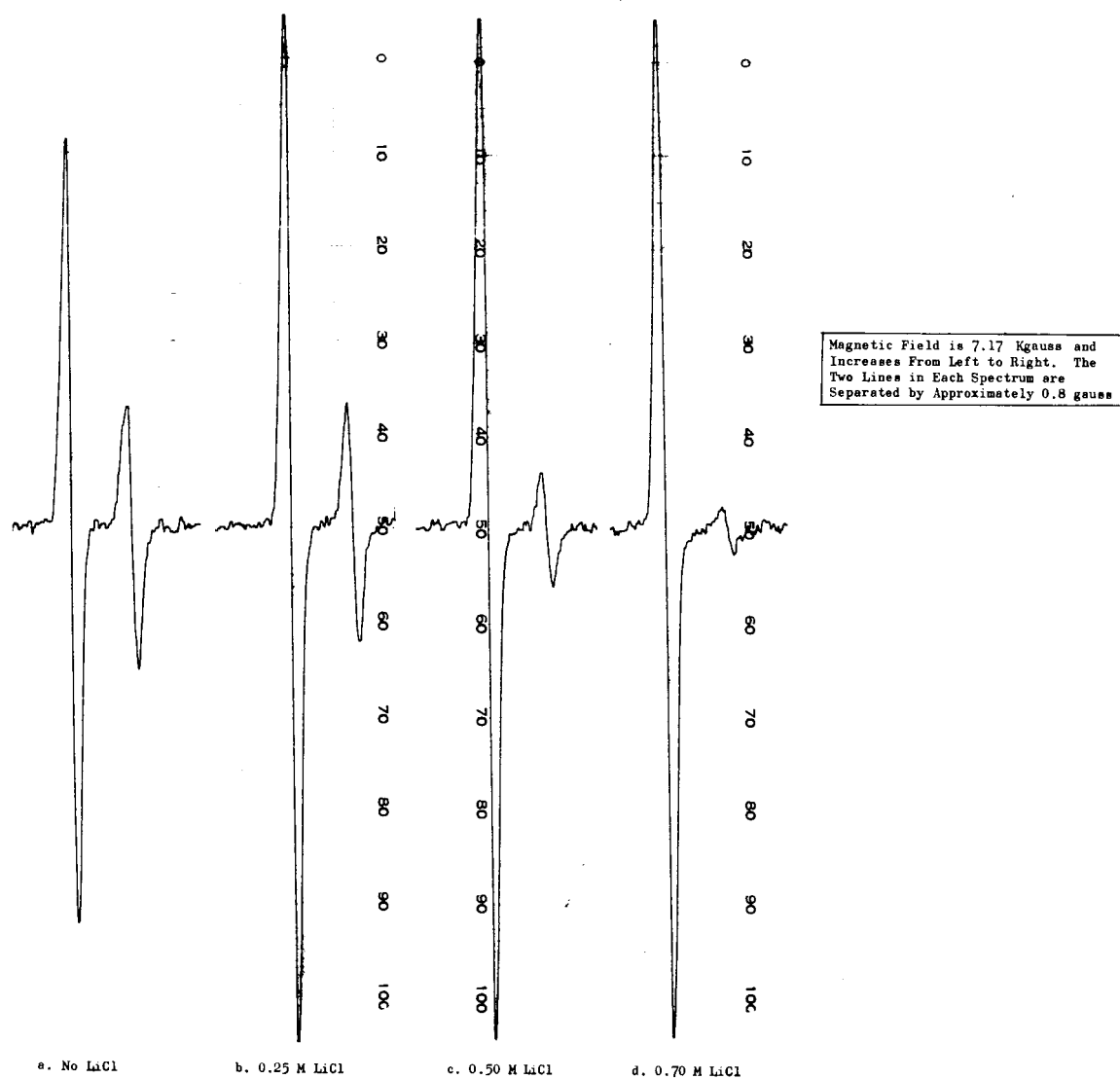
Figure 4. Al^{27} Nuclear Magnetic Resonance in 1 M AlCl$_3$/PC Containing
Various Concentrations of LiCl

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0.30

0.25

0.20

0.15

0.10

0.05

0

RELATIVE CONCENTRATION OF COORDINATING SPECIES

THE RELATIVE POPULATIONS WERE
DETERMINED FROM THE PEAKS OF
THE DERIVATIVE CURVES OF THE
SPECTRA SHOWN IN FIG. 4.

THE SOLID LINE IS A THEORETICAL
LINE BASED ON THE ASSUMPTION
THAT ALL Cl^- FORMS $AlCl_4^-$.

0   0.2   0.4   0.6   0.8   1.0

CONCENTRATION OF LiCl, MOLAR
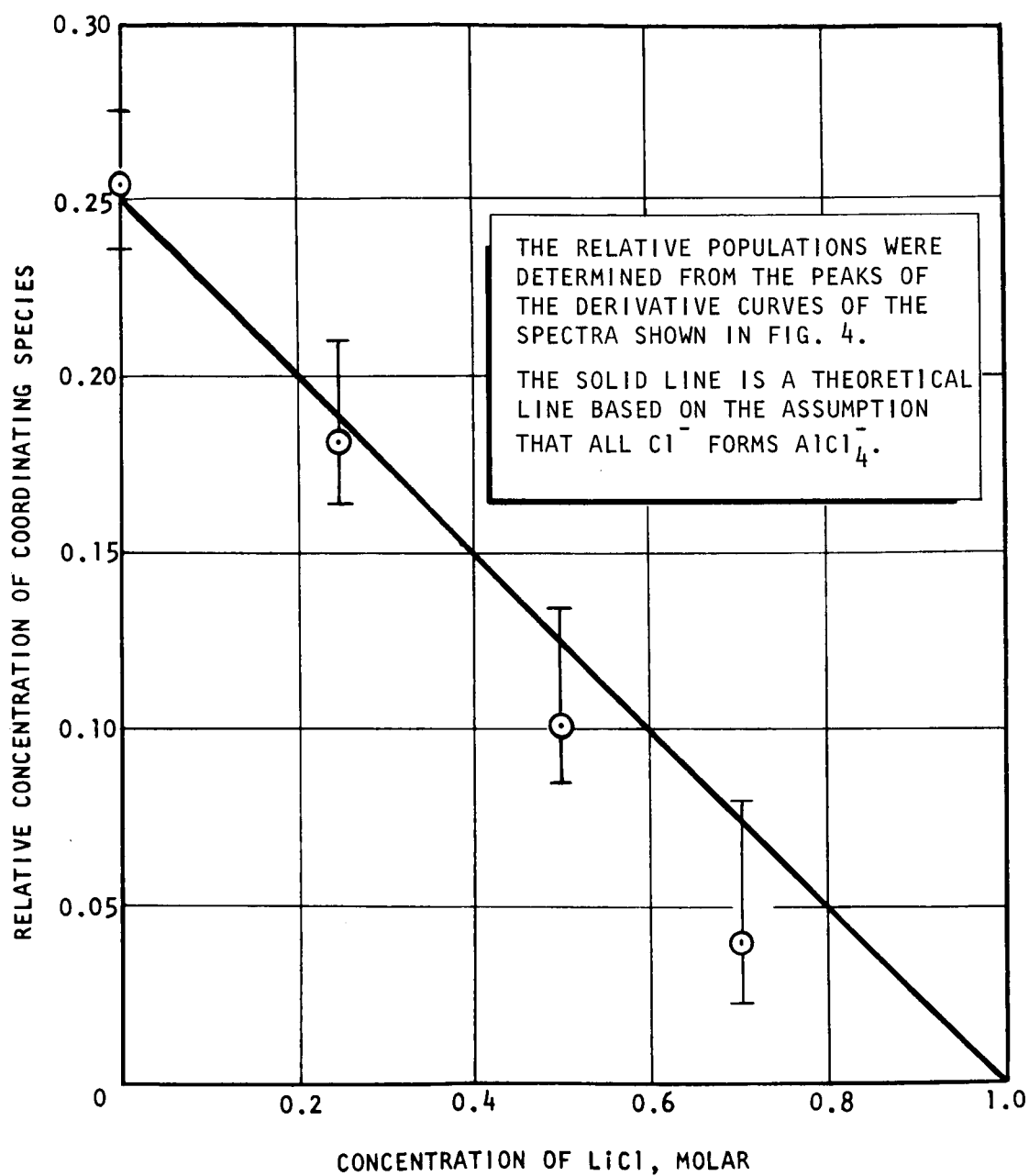
Figure 5. Approximate Relative Populations of Coordinating Al Species
in 1 M $AlCl_3$/PC as a Function of Added LiCl

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The Magnetic Field is 7.17
Kgauss and Increases From
Left to Right. The Separation
of the Two Lines is Approxi-
mately 0.8 gauss
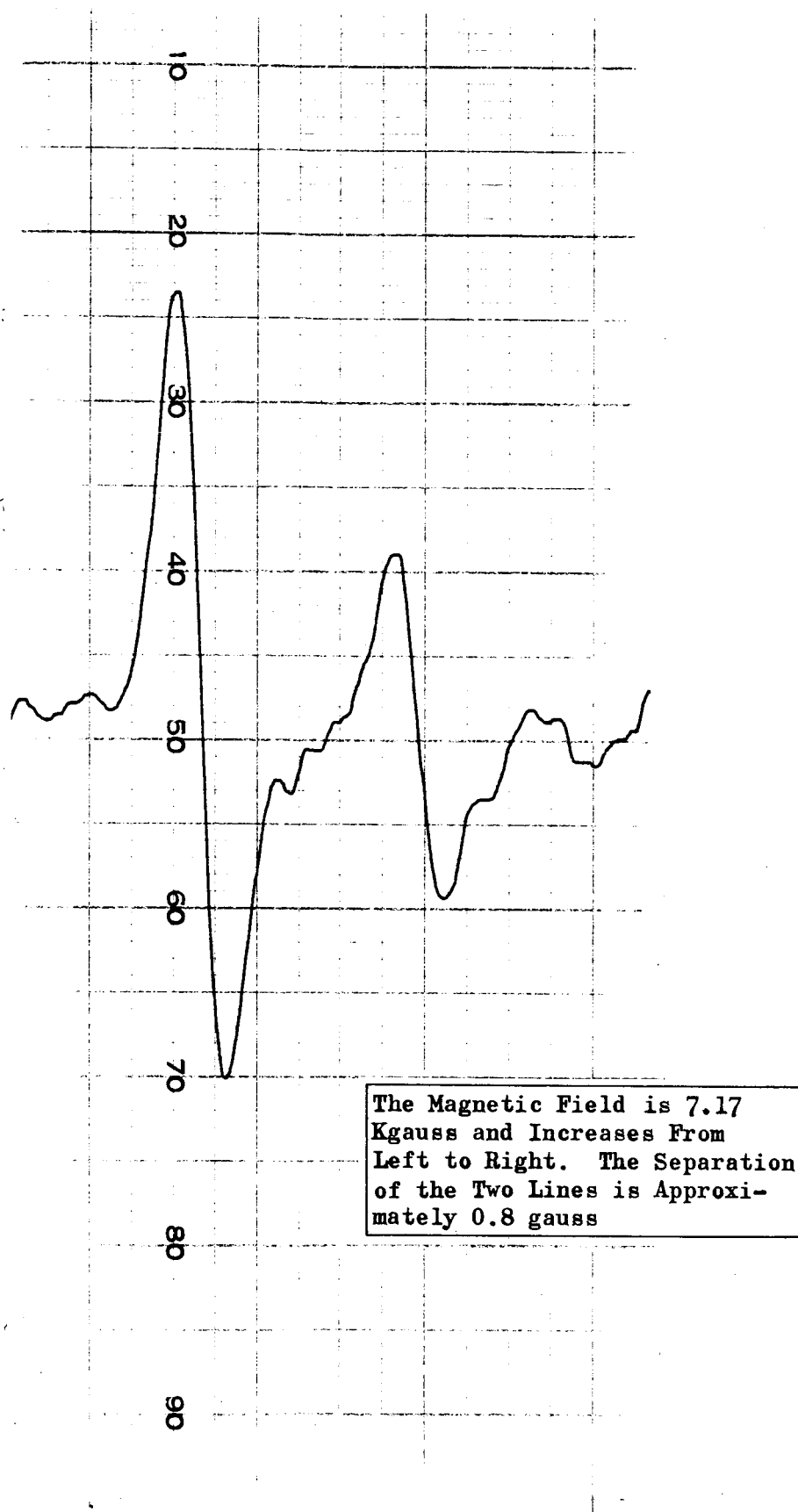
Figure   6. Al^{27} Nuclear Magnetic Resonance in 0.1 M AlCl$_3$/PC

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High Resolution H^1 NMR Spectra. The high resolution H^1 spectra of pure PC #2-6 and 1 M $AlCl_3$ #3/PC #2-6 have been shown previously (Ref. 4). The H^1 spectrum of the 1 M $AlCl_3$/PC specimen exhibits peaks due to co-ordinated PC as well as bulk PC. These peaks have been integrated to obtain the ratio of coordinated PC to coordinated plus bulk PC. Utilizing the results of the Al^{27} spectra analysis and the integration results, the coordination number for Al^{+++} in PC is 6.3. A coordination number of 6 for Al^{+++} in PC is in agreement with the coordination number of Al^{+++} in AN and other solvents (refer to previous discussion).

The spectrum of neat propylene carbonate (PC #2-12) has been studied further under high resolution conditions. The complexity of the spectrum suggests that the chemical shifts and coupling constants are quite similar in magnitude. This situation results in a very complex spectral pattern in which a simple pattern cannot be assigned to each type of resonating nuclei. However, the spectral features due to resonance of the methyl protons are well removed from the ring proton signals. For the purpose of determining coordination number, the simple pattern due to the methyl group provides all the necessary information. For the study of possible partial polymerization or degradation reaction of propylene carbonate under the influence of aluminum species, a further investigation of the spectra due to the ring protons may be advantageous.

$CuCl_2$ and LiCl in Dimethyl Formamide

The high resolution proton spectra of the solutions of $CuCl_2$ #2 in DMF #5-2 with various amounts of LiCl #2 added have been obtained over a 6-month period. The initial spectra were reported in Ref. 5 . The line positions and shapes have been observed to change continuously and apparently asymptotically over that time. In addition, the visible color of the solutions has gradually changed. The freshly prepared solutions range in color from green for no LiCl to orange for 1 and 2 M LiCl. On standing, the green solutions become reddish orange while the orange solutions remain

fairly constant. A 0.1 M solution of $CuCl_2$ #2 in DMF #5-2 was observed to yellow immediately following preparation but became colorless in a few days. An explanation of the processes occurring on this long time scale has not yet been formulated. It is presumed that the different colors arise from different copper complexes. The exact nature of the chemical environments involved and the mechanism for the conversion of copper species are still undetermined. The changes are also unknown.

Periodic monitoring of the NMR spectrum during the time period of interest has resulted in the following group of observations:

1. The spectra of freshly prepared 1 M $CuCl_2$/DMF solutions consist of two very broad lines, one from the six methyl group protons and one from the single aldehyde-type proton.
   a. Addition of LiCl results in decreased line width of both lines.
   b. The separation between the lines decreases with added LiCl.

2. All spectral features decrease in line width with time.

3. The downfield, smaller, broad line shifts toward the stronger, upfield line and asymptotically approaches 304 Hz which is the value of the splitting observed in neat DMF. This is shown in Fig. 7. With 2 M LiCl added, the initial splitting is quite close to the final value and only a slight sharpening of the line has been detected.

4. After several months, a small peak appears far downfield. The splitting increases with time and asymptotically approaches a position which is dependent on the LiCl concentration as shown in Fig. 8.

The preceding results have required a revision of the interpretation of the spectra presented in Fig. 3 of Ref. 4. The two lines found in freshly prepared 1 M $CuCl_2$/DMF appear to be due to the aldehyde and methyl protons rather than due to a distinction between coordinated and bulk DMF.
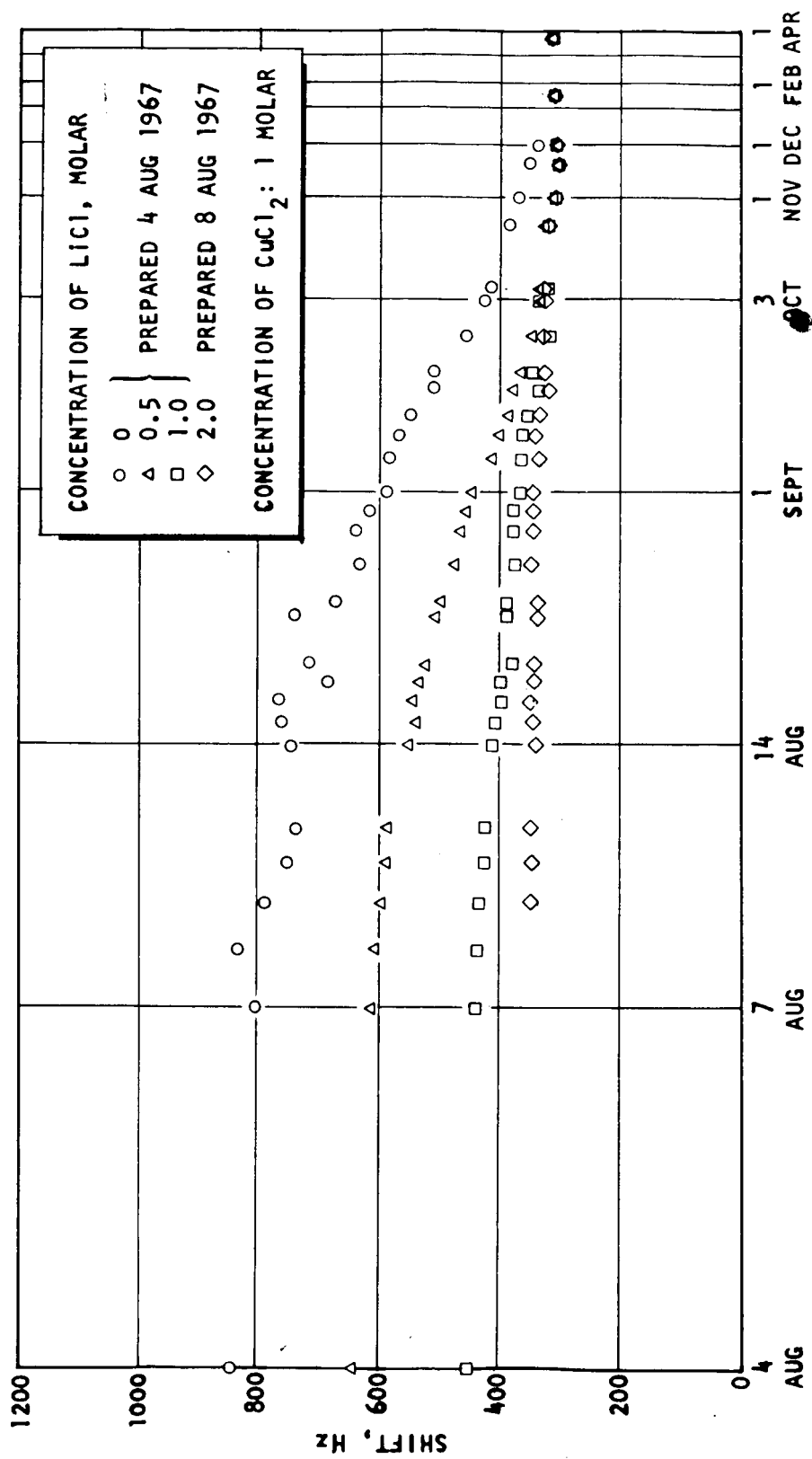
Figure 7. Splitting of Large Proton Peaks as a Function of Time for LiCl + CuCl₂/DMF
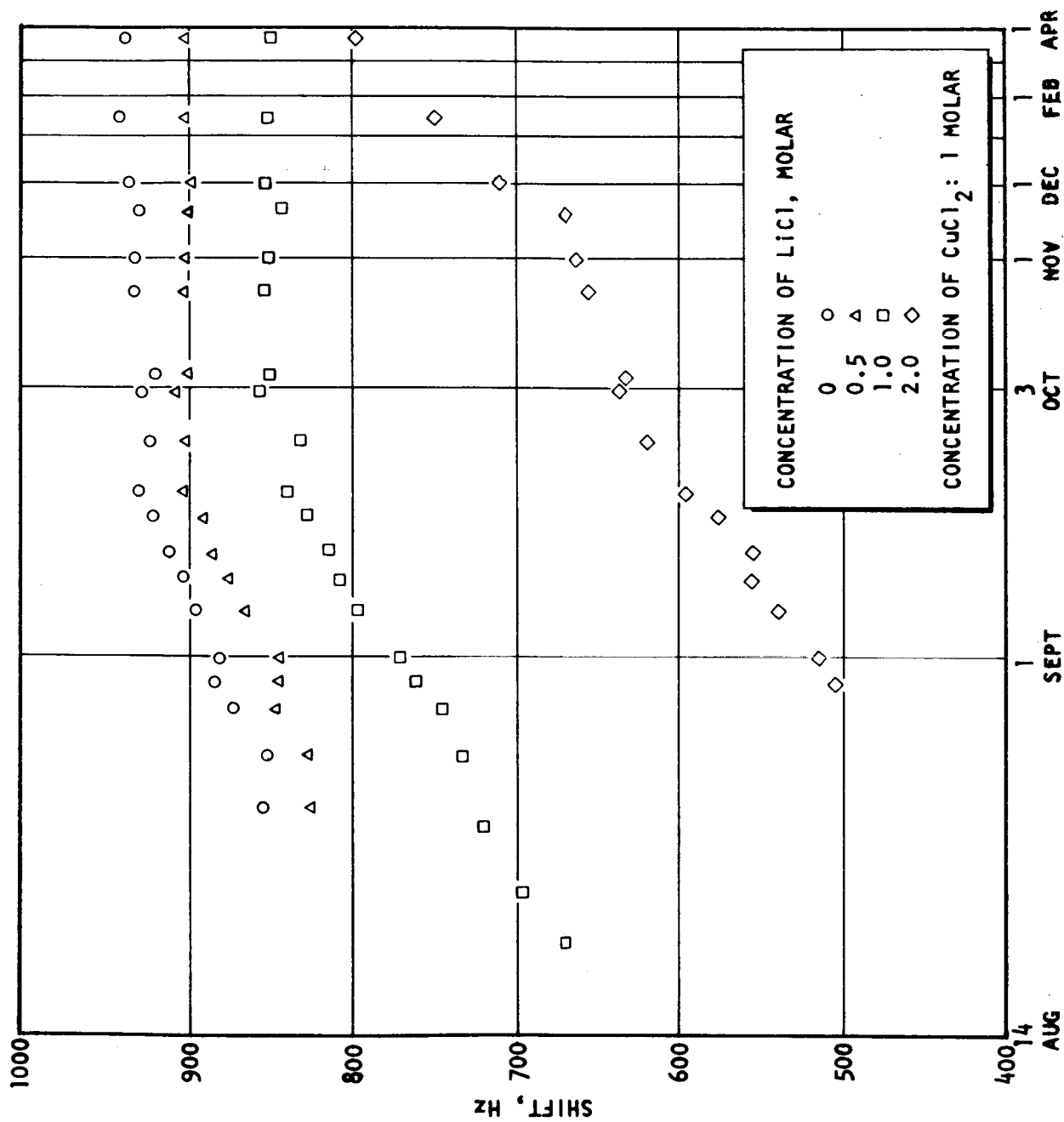
Figure 8. Position of Small Far-Downfield Resonance as a Function of Time for LiCl + CuCl$_2$/DMF

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These observations suggest that the solvent is initially rapidly exchanging in the solvation sphere of a paramagnetic species. The changes in position and line shape are consistent with the formation of a species in which the solvent is shielded from the paramagnetic electron. Evidence that this shielding is likely due to the chloride ion is provided by the smaller shifts observed with added LiCl. The smaller shift occurs both initially in the broad line displacement and in the position of the small peak far downfield in the later spectra.

Broadline H^1 and Li^7 spectra have also been recorded in some of these specimens as a function of radio-frequency power 2 weeks after preparation. The purpose of these experiments was to demonstrate the effect of the paramagnetic species on the relaxation times of these resonances. As expected, the saturation behavior of the H^1 resonances in DMF containing LiCl and $CuCl_2$ differs from that in neat DMF. The line saturates less readily in the DMF containing $CuCl_2$ as a result of increased relaxation via interaction of protons with the spin of the paramagnetic species. However, the saturation behavior of the Li^7 line in 1 M LiCl + 1 M $CuCl_2$/DMF is the same as it is in 1 molar lithium chloride solution (LiCl #2/DMF #5-2). This suggests that the relaxation of the Li resonance is not affected by the paramagnetic species. Thus far, this result is not explained. It may be due to "shielding" of the Li^+ ions from the paramagnetic species by coordinated molecules surrounding the paramagnetic species, and/or molecules coordinated by the Li^+ ions, or because the paramagnetic copper species have structures similar to $CuCl_n^{-(n-2)}$ with the paramagnetic electron in the center of the structure. The high resolution H^1 spectra of 1 M LiCl/DMF do not show coordinated peaks; however, Li^+ ions are expected to coordinate less strongly than Al^{+++} ions resulting in a much higher exchange rate. High exchange rates would preclude the observation of coordinated peaks at room temperature.

PHYSICAL PROPERTY DETERMINATIONS

Solubility Measurements

The solubility data collected during this report period are presented in
Table 4. The samples were prepared basically in the manner described in
Ref. 3. Copper contents of the samples diluted with water were measured
by atomic absorption. The data presented represent,therefore, the solubility
of copper in these solutions and do not yield any information concerning the
fate of the fluoride ions.

The solubility of CuF_2 in the pure solvents DMF and PC was very low. It
was increased to a great extent by the addition of $LiClO_4$ or LiCl. This
can be explained by the precipitation of LiF according to

$$CuF_2 + 2\ LiCl = CuCl_2 + \underline{2\ LiF} \tag{4}$$

and

$$CuF_2 + 2\ LiClO_4 = Cu(ClO_4)_2 + \underline{2\ LiF} \tag{5}$$
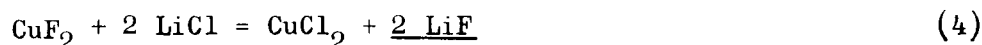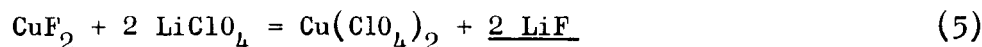
No data are available for the solubility of copper perchlorate. Copper
chloride has a high solubility in DMF, and it is significant that the
solubility of CuF_2 in 1 M LiCl/DMF does not appear to depend on the
temperature and fulfills the stoichiometric requirements of Eq. 4. The
solubility of CuF_2 in 1 M LiCl + 0.075 M $AlCl_3$/DMF appears to be signi-
ficantly lower than in 1 M LiCl/DMF. An explanation cannot be given very
readily, although a precipitation of a species containing copper and
aluminum may be a possibility.

The solid residue obtained by preparing the saturated solution of CuF_2
in 1 M LiCl/DMF was examined as shown schematically in Fig. 9. One gram
of CuF_2 #3 was added to 15 milliliters of 1 M LiCl #2/DMF #5-1. The solid
portion of the resulting mixture was collected and analyzed; it contained
1.53 mmoles of copper (as much copper as contained in 0.16 gram of CuF_2)
and 14.7 mmoles lithium, probably present in the form of LiF. Considering
the limited accuracy of the procedures involved, the precipitation of LiF
according to Eq. 4 appears to be quantitative.

TABLE 4

SOLUBILITY DETERMINATIONS

Solute	Solvent	Temperature, C	Color	Solubility, molar
CuF_2 #3	PC #2-11	25	Colorless	2×10^{-4}
		60	Colorless	4×10^{-4}
CuF_2 #3	1 M $LiClO_4$ #2/PC #2-11	25	Light green	4.7×10^{-3}
		60	Light green	6.3×10^{-3}
CuF_2 #3	DMF #5-6	25	Colorless	1×10^{-4}
		60	Colorless	2×10^{-4}
CuF_2 #3	1 M LiCl #2/DMF #5-1	25	Yellow-brown	0.53
		60	Yellow-brown	0.52
CuF_2 #3	1 M LiCl #2 + 0.075 M $AlCl_3$ #3/DMF #6-2	25	Yellow-brown	0.44
		60	Yellow-brown	0.44
$CuCl_2$ #3	1 M LiCl #2 + 0.075 M $AlCl_3$ #3/DMF #6-2	25	Dark red-brown	1.89

START:

15 MILLILITERS
1 M LiCl/DMF
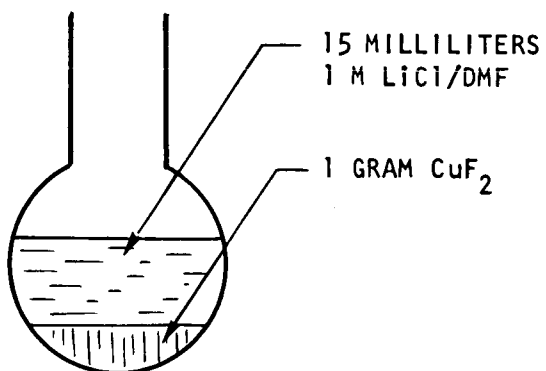
1 GRAM CuF_2

ANALYSIS:

ASSUMED TO BE PRESENT:

15 MILLILITERS 0.53 M Cu
(CORRESPONDING
TO 0.81 GRAM CuF_2)

0.5 M $CuCl_2$/DMF

LiF (+ SURPLUS CuF_2)

1.53 MMOLES Cu
(CORRESPONDING TO
0.16 GRAM CuF_2)

14.7 MMOLES Li
(AS COMPARED TO 15
MMOLES Li IN ORIGINAL
SOLUTION)

$$CuF_2 + 2\ LiCl = CuCl_2 + \underline{2\ LiF}$$
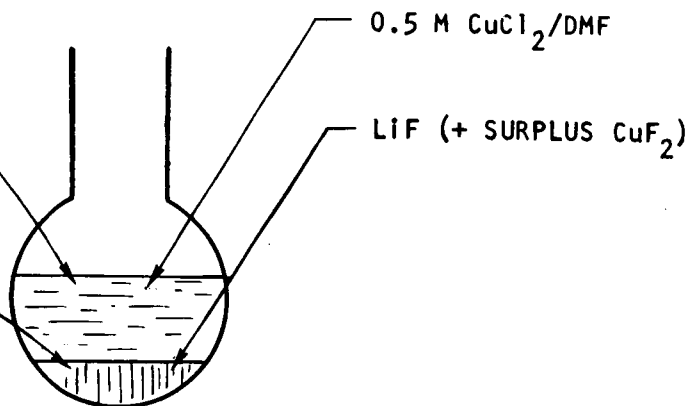
Figure 9. Examination of Precipitate Obtained in
Dissolving CuF_2 in 1 M LiCl/DMF

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The solubility of $CuCl_2$ in 1 M LiCl + 0.075 M $AlCl_3$/DMF was higher than found previously in pure DMF (1.30 M at 25 C, according to Ref.3) and in 1 M LiCl/DMF (1.04 at 25 C, according to Ref.3). It appears that a certain nonreproducibility rather than a real effect exists; this is not unexpected considering the long-term changes observed in $CuCl_2$/DMF samples, because the preparation of the samples involved treatment for prolonged periods under somewhat varying conditions.

Heat of Solution

The LKB calorimeter was checked out and calibrated. Values reproducible to within less than 1/2 percent were obtained for the calorimeter constant, and heat of solution values agreeing well with those in the literature have been measured (during another program) for the dissolution of KCl in water. The heat produced by the breakage of the sample ampoule was found to be negligible.

Measurement of Vapor Pressures

A gas-saturation method as described in Ref.17 was used to measure vapor pressures. The apparatus is shown in Fig. 10. Nitrogen is bubbled through the saturator which is immersed to the level of the glass wool wad into a constant-temperature mineral oil bath. The nitrogen is saturated with the solvent, and the solvent is collected in a liquid nitrogen trap. The connection between saturator and trap is maintained above the temperature of the constant-temperature bath, if necessary by means of heating tapes. The gas flow is measured by a soap bubble flowmeter. To minimize the error caused by evaporation of water in the flowmeter, a 2:1 mixture of glycol and glycerol containing approximately 7-1/2 percent of Ultrawet 60L was used. Typical nitrogen flowrates were 1 to 2 ml/sec.

The vapor pressure data obtained to date are listed in Table 5 . Although further checks on the reproducibility of the method shall be made, the

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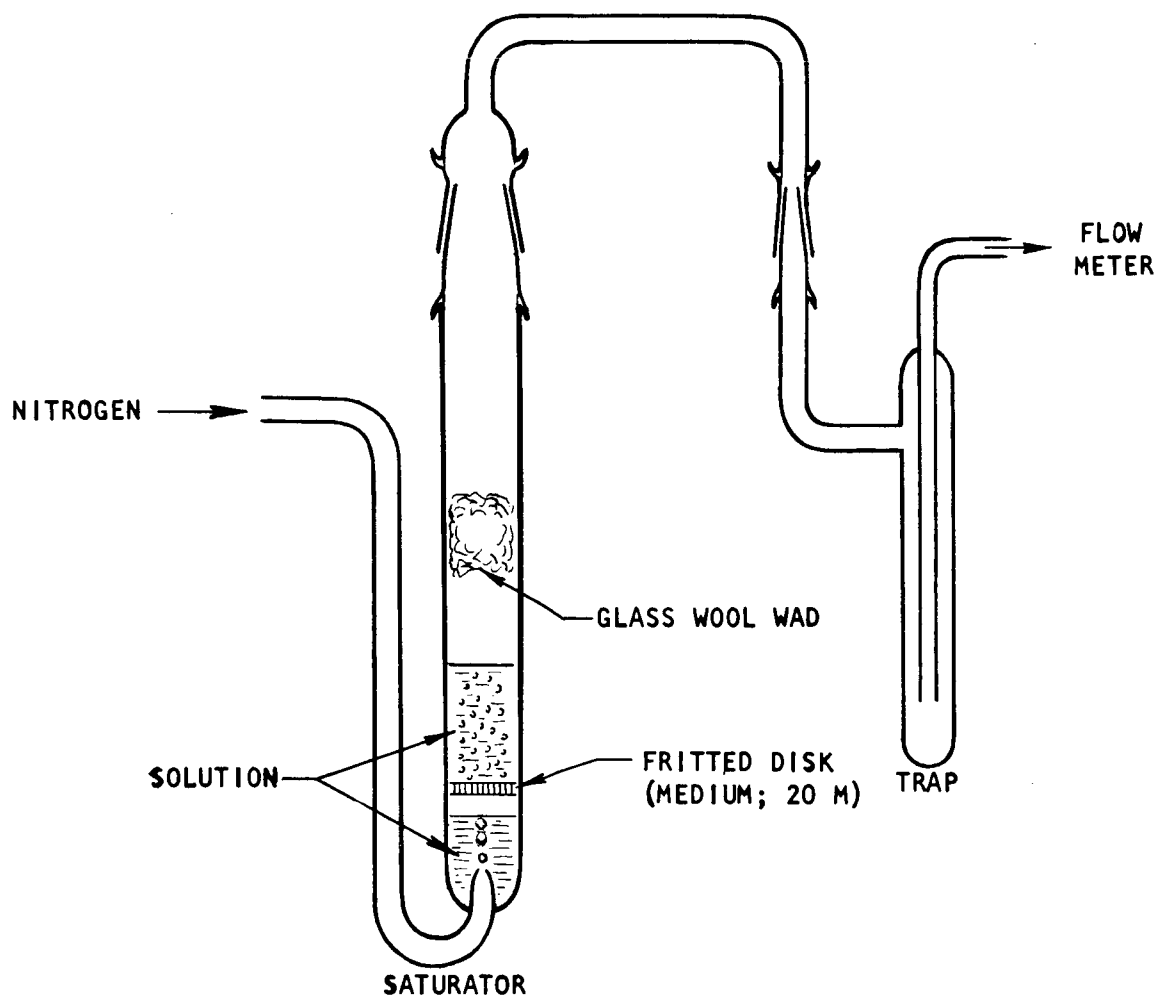
Figure 10. Apparatus for Measuring Vapor Pressures by
Gas Saturation Method

TABLE 5

VAPOR PRESSURES

Solution	Temperature, C	Vapor Pressure, mm Hg	Average Vapor Pressure Values, mm Hg
PC #2-12	25	0.082	
PC #2-12	25	0.056	
PC #2-11	60	0.803	0.80
PC #2-11	60	0.796	
1.00 M $LiClO_4$ #2/PC #2-11	60	0.657	0.66
1.00 M $LiClO_4$ #2/PC #2-11	60	0.663	
0.125 M $TMA \cdot PF_6$ #1/PC #2-11	25	0.062	
	60	0.52	
DMF #5-2	25	3.96	
DMF #5-2	25	4.10	3.95
DMF #5-5	25	3.79	
DMF #5-2	60	26.7	26.3
DMF #5-5	60	25.9	
1.00 M $LiClO_4$ #2/DMF #5-1	25	3.18	3.16
1.00 M $LiClO_4$ #2/DMF #5-1	25	3.14	
1.00 M $LiClO_4$ #2/DMF #5-1	60	22.6	
1.00 M LiCl #2/DMF #5-1	60	23.55	
1.00 M LiCl #2 + 0.075 M $AlCl_3$ #3/DMF #6-2	25	3.32	
	60	22.80	
AN #4-2	25	89.0	
1.00 M $LiClO_4$ #2/AN #4-2	25	79.4	

results generally appear to be accurate within 1 to 2 percent. Results obtained with propylene carbonate at 25 C vary to a greater extent and no average value is yet considered representative; in these experiments, the vapor pressure was very low and the amount of solvent collected was small, thus increasing experimental errors. However, the vapor pressure of AN is high, and this result will be rechecked to verify that complete saturation was obtained.

Measurement of Diffusion Coefficients by the Porous Disk Method

Diffusion coefficients are being determined by the method of Wall (Ref. 18 through 20). The procedure involves filling an evacuated porous porcelain disk with the solution to be studied, and then suspending the disk from one arm of an analytical balance on a fine wire, in a large volume of pure solvent. Measurement of the apparent weight change of the disk as a function of time allows the determination of the diffusion coefficient from the equation:

$$\log \left[W(t) - W(\infty) \right] = -\alpha\, Dt + b \tag{6}$$

where $W(t)$ is the apparent weight of the suspended disk at time t, $W(\infty)$ is the weight after equilibrium has been reached, D is the diffusion coefficient, and α and b are constants.

Calibration runs with a solution of known diffusion coefficient allow the determination of the apparatus constant, α. Apparatus constants were determined using standard 1.5 M KCl/H_2O - H_2O ($D = 1.87 \times 10^{-5}$ cm^2 sec^{-1} at 25 C; Ref. 21) for two recommended Micro-Porous Filter Disks (2 by 1/4 inch, No. 10 porosity), obtained from Silas Flotronics. The average values for the constants of the two frits to be used were $\alpha_1 = 6.49$ cm^{-2} and $\alpha_2 = 6.59$ cm^{-2}, respectively.

The diffusion coefficients determined are presented in Table 6.

TABLE 6

DIFFUSION COEFFICIENTS, D, OF DIMETHYL FORMAMIDE ELECTROLYTES AT 25 C

Solution	Solvent	Diffusion Coefficient, cm^2 sec^{-1}	Diffusion Coefficient Average Value, cm^2 sec^{-1}
1.00 M LiClO$_4$ #2/DMF #5-1	DMF #5*	7.28×10^{-6}	7.3×10^{-6}
1.00 M LiClO$_4$ #2/DMF #5-1	DMF #5-3	7.30×10^{-6}	
1.00 M LiCl #2/DMF #6-1	DMF #6-2	5.76×10^{-6}	5.9×10^{-6}
1.00 M LiCl #2/DMF #6-2	Mixture of DMF #6-1, 6-2, and 6-4	5.99×10^{-6}	
1.00 M LiCl #2 + 0.075 M AlCl$_3$ #3/DMF #6-2	Mixture of DMF #6-3 and 6-5	5.72×10^{-6}	

*Undistilled, spectrograde material

In all five runs, plots of log $\left[W(t) - W(\infty) \right]$ vs time gave relatively straight lines indicating that Eq. 3 was being followed. Approximately 1.5 liters of solvent were used in each determination and all measurements were made at 25 ±0.02 C.

Determination of Diffusion Coefficients
by Chronopotentiometry

Chronopotentiometry is being used to determine the diffusion coefficient of copper in 1 M LiClO$_4$/DMF and 1 M LiCl/DMF. Preliminary studies have been conducted on the reduction of copper in these solutions. Figure 11 shows a chronopotentiogram for the reduction of CuCl$_2$ in 1 M LiCl/DMF. The wave for the reduction of CuCl$_2$ in 1 M LiClO$_4$/DMF is similar. The copper wave is well defined and the transition time may be determined with good precision. A tentative value for the diffusion coefficient of copper(II) in 1 M LiCl/DMF is 1.1×10^{-5} cm^2/sec. This value is
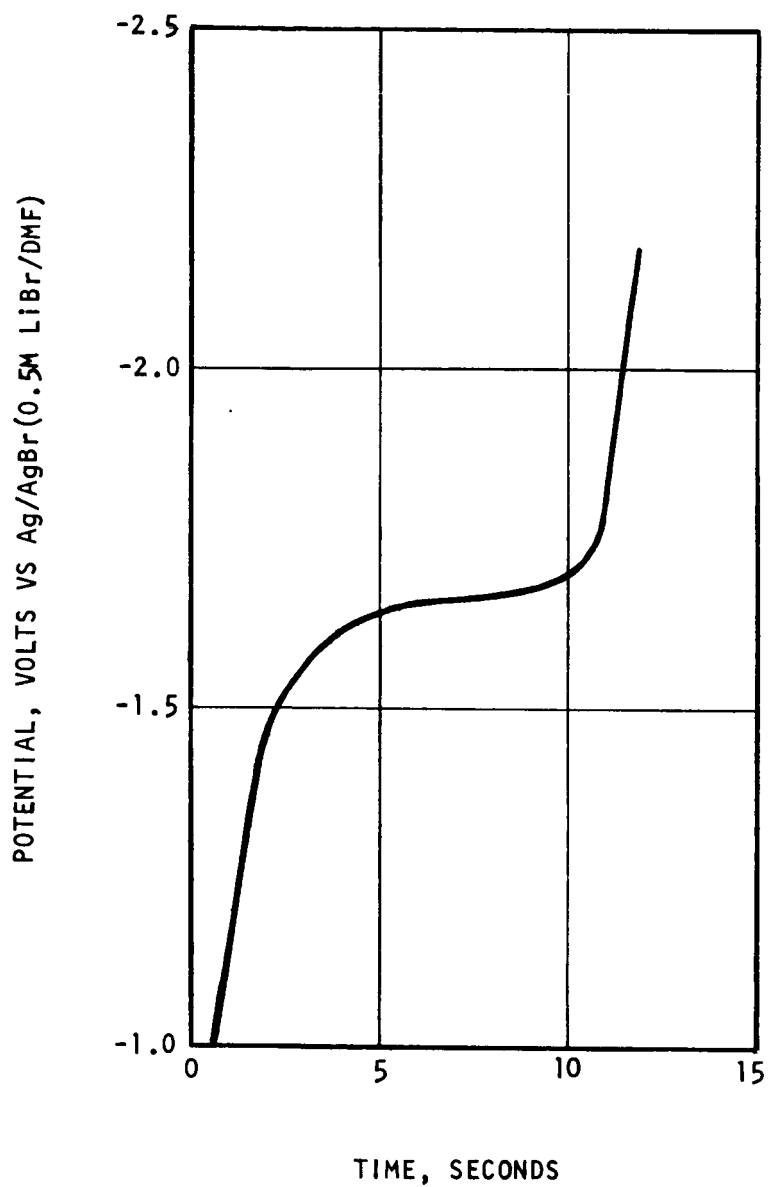
Figure 11. Chronopotentiogram of 0.01 M_2CuCl_2 in 1 M LiCl/DMF; the
Current Density is 1.6 ma/cm²

tentative because a significant amount of water may have been absorbed by the solution during this preliminary study, and this amount of water may change the diffusion coefficient.

A second wave appears at more cathodic potentials. This wave is smaller than the copper wave and represents the reduction of water. At much lower current densities, two additional waves appear at 0 and -400 millivolts. The quarter-wave potential of the first wave is the same as the open-circuit potential of a copper electrode in this solution. The waves are not due to oxygen because the solution was deaerated with nitrogen and because the reduction potential for oxygen is -200 millivolts.

The reduction of copper(II) does not appear to present any problems, and a more accurate determination of the diffusion coefficient will be made.

Measurement of Dielectric Constants

For determination of the dielectric constant, K_o , measurements made at microwave frequencies of 8.5 and 25 GHz will be extrapolated to low frequency. A somewhat modified version of a transmission method used by Harris and O'Konski (Ref. 22) for high permittivity liquids has been adopted (Ref. 4).

Microwave Method Description. The method is based on determining the phase shift and decrease in amplitude of a wave when it travels through a dielectric sample. A reference wave of fixed phase and amplitude traveling in direction T_2 to T_1 (Fig. 12) interferes with the sample wave. The resulting standing wave position (minimum) and amplitude (or VSWR)* are then measured with and without the sample in place. From the difference in minimum position, the phase shift, Σ , of the sample wave is obtained. Measurement of the VSWRs allows calculation of Q, the decrease in amplitude.

---
*Voltage standing wave ratio
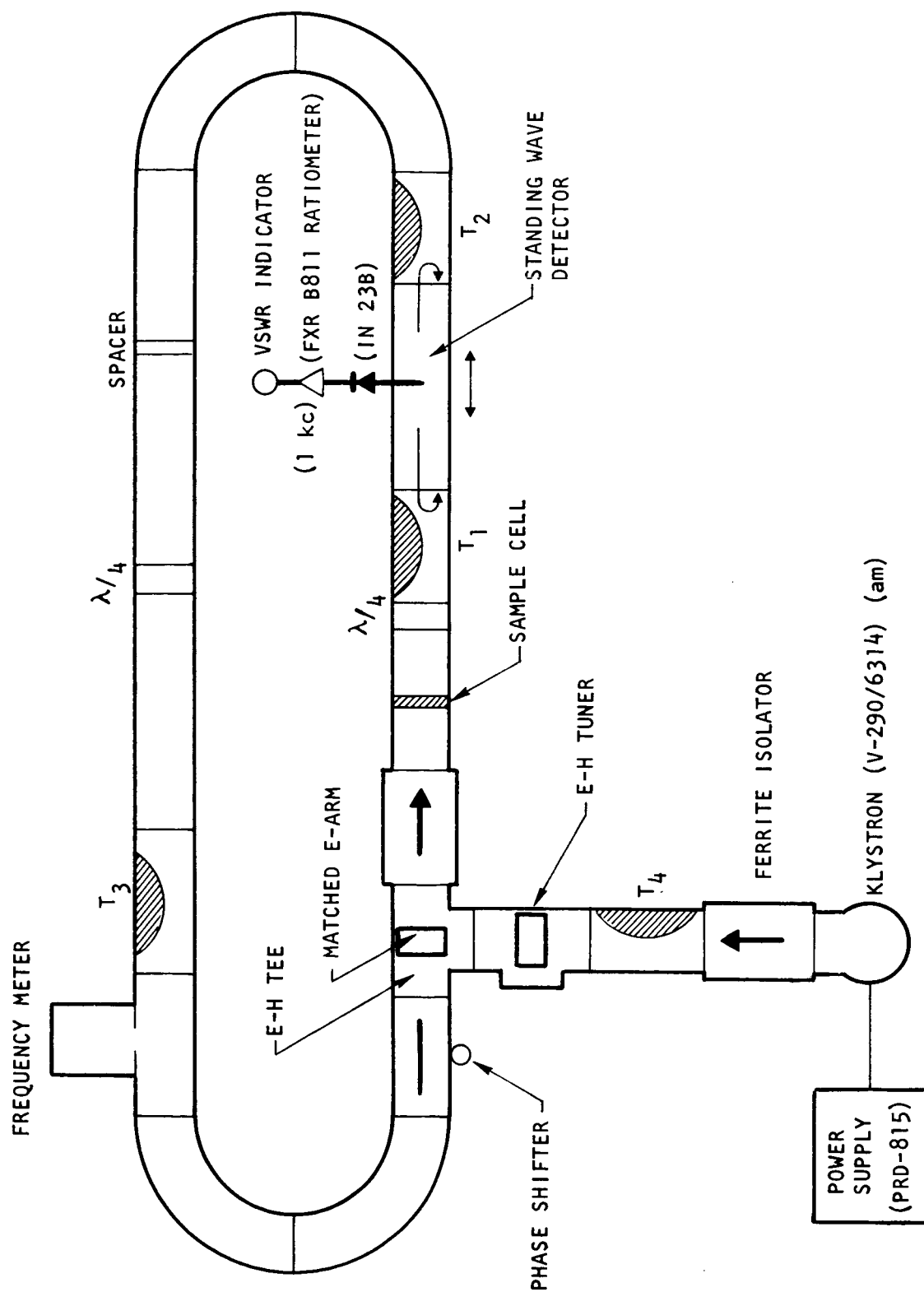
Figure 12. Microwave Setup at 8.5 GHz, $TE_{0,1}$ Mode

SPACER

$\lambda/4$

T_3

FREQUENCY METER

VSWR INDICATOR
(FXR B811 RATIOMETER)
(IN 23B)
(1 kc)

STANDING WAVE DETECTOR

T_2

T_1

SAMPLE CELL

$\lambda/4$

MATCHED E-ARM

E-H TEE

E-H TUNER

T_4

PHASE SHIFTER

FERRITE ISOLATOR

KLYSTRON (V-290/6314) (am)

POWER SUPPLY (PRD-815)

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A computer program has been used to calculate Q and Σ from assumed κ' and κ'' values ($\kappa = \kappa' - i\kappa''$). This yields graphs that show for coordinates Q and Σ a series of intersecting contours of κ' and κ'' (Ref. 22). These graphs are used for determining κ' and κ''. The computer program makes possible rapid calculation of Q vs Σ graphs for various cell widths.

The present setup (Fig. 12) differs from the apparatus described in Ref. 22. The modifications are as follows: (1) attenuators T_1 and T_2 are not fully matched (i.e., do not have reflection coefficients <0.5 percent) and (2) a ferrite isolator is used to absorb waves reflected from the filled sample cell. In Ref. 22 an iris was positioned in the reference arm to provide for cancellation of the reflected wave (Ref. 4). Both have the same purpose, that is, to prevent coupling between the two arms. Using modifi-- cation No. 2, no coupling between reference and sample waves could be detected.

Figure 12 shows only the 8.5 GHz equipment. The 25 GHz setup is similar except that the E-H tee is replaced by a directional coupler.

Corrections for Reflections. Attenuators T_1 and T_2 have small but not negligible reflection coefficients ($\sim$2.5 percent). The measured VSWRs and minimum position differences are corrected for this by subtracting the effect of the small amplitude waves reflected toward the probe (as shown by the curved arrows in Fig. 12). These calculations (Appendix A) have been also programmed on the computer. To obtain the corrections needed, the reference and the sample waves each are measured separately.

Attenuator T_1 reflects also a part of the wave transmitted through the sample. The reflected component reaches the rear face of the cell, where it is again reflected (if the cell contains liquid with a high κ') and then adds to the transmitted wave. Moving T_1 by a distance of $\lambda/4$ between two measurements and subsequently averaging the results allows cancellation of the reflected wave (Ref. 23).

A similar displacement of the isolator preceding the cell compensates for secondary reflections associated with that component.

The changes made by applying the reflection corrections depend on the κ' and κ'' values of the sample. At 8.5 GHz it has varied from -3 to +12 percent for κ' and -12 to 32 percent in κ''. Shifting the attenuator T_1 appeared to effect a less than 5 percent change in κ' and a ~ 10 percent change in κ''.

Test Results. Testing has been in part described (Refs. 4 and 5). Results obtained at 8.5 GHz for H_2O, spectrograde methanol, and AN #4-2 are presented in Table 7. Each of these was obtained by employing the quarter wavelength shift technique for cancelling reflections from T_1. The water results are within the expected $\sim \pm 5$ percent accuracy. The dielectric constant, κ', of methanol is actually lower than values for which this microwave method is best suited. Use of a thicker cell and redistillation of the methanol sample should yield better agreement. Since liquids having well known κ' and κ'' values both intermediate between those of H_2O and methanol are not available, acetonitrile has been used for testing purposes. For AN the measurements were repeated by shifting the isolator by a quarter wavelength. The results were essentially the same as in Table 7. The literature values (Ref. 27) for comparison were obtained by extrapolating the data from 30, 40, and 50 C to 24 C. Our κ' value is approximately 14 percent higher and further work is in progress using different cell thicknesses (Ref. 23) to reduce effects due to the mica windows and thereby increase the accuracy.

The apparatus for measurements at 25 GHz has not been as extensively checked out as the lower frequency apparatus. The crystal response law has not been determined as yet. Testing for coupling between the sample and reference arms has been made. No significant coupling was found. If present, such coupling would lead to large errors. Also, preliminary experiments using a rough, 0.255-centimeter-thick test cell were made on H_2O, spectrograde methanol, and AN #4-2. The κ' and κ'' obtained

(except κ' for methanol) are all within 16 percent of the literature data. Further work is being conducted on making repeat runs with attenuator (T_1) and isolator displacement to cancel out secondary reflections. Sample cells of different thicknesses will be used to improve the accuracy. The attainable accuracy may approach ± 5 percent also with this setup.

TABLE 7

DIELECTRIC CONSTANT TEST RESULTS AT 8.5 GHz (24 C)

Sample	d, cm	ν, GHz	κ'	κ''	Literature Values		Reference
					κ'	κ''	
H_2O	0.383	8.49	63.9	29.2	64.9	27.5	24
MeOH	0.383	8.49	8.05	9.8	8.85	8.95	25, 26
AN #4-2	0.383	8.49	35.6	9.0	31.3	8.2	27

APPENDIX A

REFLECTION CORRECTIONS FOR DIELECTRIC CONSTANT MEASUREMENTS

$$E_1(x) = R_0 e^{i(1/2)x} - R_1 R_0 e^{-i(1/2)x}$$

$$E_2(x) = e^{i\varphi_2}\left(e^{-i(1/2)x} - R_2 e^{-ix_2} e^{i(1/2)x}\right)$$

$$E_2'(x) = e^{i\varphi_6} RR_0 \left(e^{-i(1/2)x} - R_2 e^{-ix_2} e^{i(1/2)x}\right)$$

$$E_{3,min} = E_1(x_3) + E_2(x_3)$$

$$E_{3,max} = E_1(x_3+\pi) + E_2(x_3+\pi)$$

$$E_{4,min} = E_1(x_4) + E_2'(x_4)$$

$$E_{4,max} = E_1(x_4+\pi) + E_2'(x_4+\pi)$$

The reflection coefficients R_0 and R are calculated by successive approximations from the measured standing wave ratios:

$$(VSWR)_3 = \left|E_{3,max}\right| \Big/ \left|E_{3,min}\right| \quad \text{and} \quad (VSWR)_4 = \left|E_{4,max}\right| \Big/ \left|E_{4,min}\right|.$$

The phases φ_2 and φ_6 are obtained from

$$\frac{d}{dx_3}\left(\left|E_{3,min}\right|\right)^2 = 0 \quad \text{and} \quad \frac{d}{dx_4}\left(\left|E_{4,min}\right|\right)^2 = 0, \text{ respectively.}$$

The amplitude decrease (Q) is RR_0 whereas the phase shift (Σ) is $\varphi_2 - \varphi_6$. These are then used to read off κ' and κ'' from the computed graphs.

The symbols are:

R = reflection coefficients; R_0 for composite wave E_3 and R for E_4

E = electric fields for the waves $\left(E'\right.$ refers to transmitted sample wave$\left.\right)$

Subscripts

1 = reference wave

2 = sample wave

3 = composite wave with empty cell

4 = composite wave with filled cell

x = distances $\left(2\pi z/\lambda g\right)$ with subscripts designating the minima of the corresponding standing waves, i.e. x_3 is minimum of wave with empty cell.

WORK PLANNED FOR NEXT QUARTER

PREPARATION OF ELECTROLYTES

Work on this phase will be performed essentially on a routine basis.

STRUCTURAL STUDIES

Some work will be performed on the $LiCl + AlCl_3/DMF$ system and on dilute $LiCl + AlCl_3/AN$. Most of the effort will be devoted to DMF systems containing LiCl and copper halides, utilizing both EPR and NMR. It is planned to initiate work on the $TMA \cdot F + BF_3/PC$ and $TMA \cdot F + BF_3/DMF$ systems.

PHYSICAL PROPERTY DETERMINATIONS

Determinations of physical properties will continue. Solubility, conductance, sonic velocity, vapor pressure, diffusion coefficient, and transference measurements will be supplemented. The first heat of solution values and dielectric constants will be determined.

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Expenditures (March 22, 1968)

Contract No. NAS 3-8521

Prepared for

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Cleveland, Ohio

APPROVED: _____
K. H. Mueller
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from February 19 to March 22, 1968 were:

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$17,787

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13. ABSTRACT

Studies of aprotic electrolytes based on three solvents: propylene carbonate, dimethyl formamide, and acetonitrile, were continued. Characterized components were used to prepare the electrolyte solutions. Structural studies of electrolytes containing lithium chloride, aluminum chloride, and cupric chloride were performed utilizing nuclear magnetic resonance. The physical properties studied include solubilities, vapor pressures, diffusion coefficients, and dielectric constants.

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Dimethyl Formamide						
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